

(19) World Intellectual Property
Organization
International Bureau



10/5/04



(43) International Publication Date
29 April 2004 (29.04.2004)

PCT

(10) International Publication Number
WO 2004/035725 A1

(51) International Patent Classification⁷: **C11D 11/00**, (74) Agents: **BROWN, Andrew, Stephen et al.**; Reckitt Benckiser plc, Group Patents Department, Dansom Lane, Hull, Yorkshire HU8 7DS (GB).
17/04, 1/00, 3/37, A47L 11/34

(21) International Application Number:
PCT/GB2003/004329

(22) International Filing Date: 6 October 2003 (06.10.2003)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
0223848.3 12 October 2002 (12.10.2002) GB

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

(71) Applicant (*for all designated States except US*): **RECKITT BENCKISER N.V.** [NL/NL]; Kantoorgebouw De Appelaer, De Fruittuinen 2-12, NL-2132 NZ Hooddorp (NL).

(71) Applicant (*for MN only*): **RECKITT BENCKISER (UK) LIMITED** [GB/GB]; 103-105 Bath Road, Slough, Berkshire SL1 3UH (GB).

(72) Inventors; and

(75) Inventors/Applicants (*for US only*): **DE DOMINICIS, Mattia** [IT/IT]; Reckitt Benckiser Italia, Piazza S. Nicolo 12/3, I-30034 Mira VE (IT). **RIGHETTO, Zefferino** [IT/IT]; Reckitt Benckiser Italia, Piazza S. Nicolo 12/3, I-30034 Mira VE (IT).

Declaration under Rule 4.17:

— *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii)) for all designations*

Published:

— *with international search report*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: CARPET CLEANING COMPOSITION

(57) Abstract: The invention relates to a water-soluble product comprising a water-soluble polymer encasing a liquid carpet cleaning composition.

WO 2004/035725 A1

CARPET CLEANING COMPOSITION

The invention relates to a water-soluble product comprising a water-soluble polymer encasing a liquid carpet cleaning composition.

Carpet cleaning machines are becoming increasingly popular purchases for the home as the cost of such appliances falls. However, it is more common to hire these types of machines. The long periods between cleaning inevitably means that the accumulated dirt in the carpet requires a specialised carpet cleaning formulation.

The encasing polymer can be made of any water soluble or water dispersible polymer which can be sealed. Examples of water-soluble polymers are Poly(vinylalcohol) (PVOH), cellulose derivatives such as hydroxypropyl methyl cellulose (HPMC), gelatin, poly(vinylpyrrolidone), poly(acrylic acid) or an ester thereof or poly(maleic acid) or an ester thereof. Copolymers of any of these polymers may also be used.

The following are some advantages found in this invention:

- A pre-dosed carpet cleaning composition.
- The pre-dosed products prepared have high storage stability, up to 6 months at 50°C, even if the liquid contains water, at total levels of up to 50% wt.
- The liquid compositions have higher soil removing performance compared to current Resolve™ for steam

machine carpet cleaning liquid, taken as a reference.

The products of the invention need to be soluble in water and to dissolve in a short period, typically less than 5 minutes, without any stirring, to produce a product having low or no solid residue. A common feature of many carpet cleaning machines is the presence of a fine mesh filter preventing solid material from entering and damaging the internal mechanisms. Therefore, it is important that there are no/low solid residues in the reservoir so as to avoid any blockages.

We present as a feature of the invention a method of cleaning a soiled carpet the method comprising adding to the reservoir of a carpet cleaning machine a water-soluble product comprising a liquid carpet cleaning composition encased in a water-soluble polymer, adding water to the reservoir of the carpet cleaning machine prior to or after adding the product, and operating the carpet cleaning machine on the soiled carpet.

The pre-dosed composition can be added to the reservoir of the carpet cleaning machine either before or after the addition of water, preferably it is added prior to the addition of water.

The term "water-soluble" is taken to include water dispersible.

Preferably the liquid carpet cleaning composition comprises less than 5%w/w of free water, preferably less than 4, 3, 2 or 1 %w/w. Even more preferably the composition is anhydrous.

As used herein the term "free water" as defined in WO 02/16222, incorporated herein by reference. There is no direct correlation between the actual amount of water present in a composition and the amount of free water.

5 Free water does not include water which is not available to the surrounding water-soluble polymer such as water held within a gelled matrix or water of solvation of any components present in the composition.

10 In order to determine the amount of free water present in a composition, a standard loss-on-drying determination test may be carried out. A sample of the composition, usually 10 g, is weighed, and then maintained at 60°C for 3 hours under a partial vacuum of
15 200 mbar (20 kPa). The sample is then re-weighed, and the weight lost determined. In the present invention, the loss on drying of the composition is preferably less than 5 wt%, preferably less than 4, 3, 2 or 1 %w/w. Even more preferably the composition is anhydrous.

20 The actual amount of water present in the composition may be in excess of the amount of free water, as defined above, since the total water content includes water of solvation and water held within a gelled matrix.
25 The total amount of water in the composition is, for example, more than 5, 10, 15, 20, 25 or 30%w/w. Total water levels of up to 50%w/w are preferred. The total water content may be less than 80% w/w, for example less than 70, 60, 50, 40, 30%w/w.

30

In an alternative embodiment the free water content may be greater than 5%w/w by the inclusion of sufficient components in the composition to raise the ionic strength of the composition this may be achieved by the use of

suitable compositions, such as electrolytes, that prevent the water being available to the water-soluble polymer, see WO8904282 and EP0518689.

5

The product provides the consumer with a convenient form, which is pre-dosed, avoiding the need to dilute the product before it is added to the machine.

10 We have developed a pre-dosed product that contains a composition for carpet cleaning extraction machines. These compositions comprise at least one builder, one surfactant and a super wetting agent. Preferably these compositions additionally comprise up to 10% w/w of one
15 or more of the following optional ingredients, antifoaming agent, solvent, fragrance, preservative, thickener, dye, bactericide and filler. Each optional ingredient may be present in an amount of up to 3%w/w.

20 The superwetting agent can be added to overcome the problem associated with the high repellancy of carpet to water. This is primarily caused from two sources, the first being the amount of soiling which can accumulate on carpets and, secondly, the prevalence of stain repelling
25 treatments, which are increasingly commonly applied to carpets either during manufacture or by the consumer. In this invention a super wetting agent is a special surfactant added at levels of below 10%w/w of the composition, preferably below 5% w/w, of the composition,
30 that can, combined with any other surfactant present in the composition, is able to lower the surface tension of the final diluted liquid cleaning formulation to values below 28mN/m, when 10g is dissolved in 4 litres of water.

A problem associated with cleaning carpets is the high repellancy of the carpet to water. This is primarily caused from two sources, the first being the amount of soiling which can accumulate on carpets and, 5 secondly, the prevalence of stain repelling treatments which are increasingly commonly applied to carpets either during manufacture or by the consumer. In this invention a "super wetting" agent is added to the composition and is a special surfactant added at levels of below 10%w/w 10 of the composition, preferably below 9, 8, 7, 6 or 5% w/w, of the composition. It is preferred that at the levels described above, and in combination with any other surfactant present, it is able to lower the surface tension of the final diluted liquid cleaning formulation 15 to value below 28 mN/m, when 10g is dissolved in 4 litres of water.

We present as a further feature of the invention a water-soluble product comprising a carpet cleaning 20 composition encased in a water-soluble polymer (ideally the carpet cleaning composition comprising at least one surfactant and at least one super wetting agent wherein the combined surface tension effect of the surfactant and the super wetting agent in the composition is capable of 25 reducing the surface tension of water below 28 mN/m when 10g of the composition is dissolved in 4 litres of water.

Preferably a product is used having from 1 to 25g of liquid carpet cleaning composition per machine, ideally 30 from 2 to 12g, and preferably from 3 to 10g.

Builders

The carpet cleaning composition comprises at least one builder active or a combination of builders from 1 to 90%w/w, preferably from 65 to 85%w/w. Preferably the builder is soluble or miscible with the liquid carpet
5 cleaning composition.

Suitably carboxylate compounds are used and include the monomeric polycarboxylates, or their acid forms and polymeric polycarboxylic acids or their salts. Polymeric
10 polycarboxylic acids are preferred for the reasons given above, in terms of protecting the water-soluble polymer.

The carboxylate builder can be monomeric or polymeric in type, monomeric polycarboxylates are
15 generally preferred for reasons of cost and performance.

Suitable and preferred polymeric polycarboxylic acids are iminosuccinic acid or polyaspartic acid, mixtures thereof or their metal/amino salts. Examples of
20 these polymers are Baypure CX 100/34 and Baypure DS 100/40 supplied from Bayer.

Suitable carboxylates containing one carboxy group include the water soluble salts of lactic acid, glycolic
25 acid and ether derivatives thereof. Polycarboxylates containing two carboxy groups include the water-soluble salts of succinic acid, malonic acid, (ethylenedioxy) diacetic acid, maleic acid, diglycolic acid, tartaric acid, tartronic acid and fumaric acid, as well as the
30 ether carboxylates and the sulfinyl carboxylates. Polycarboxylates containing three carboxy groups include, in particular, water-soluble citrates, aconitrates and citraconates as well as succinate derivatives such as the carboxymethloxysuccinates described in GB-A-1,379,241,

lactoxysuccinates described in GB-A-1,389,732, and aminosuccinates described in NL-A-7205873, and the oxypolycarboxylate materials such as 2-oxa-1,1,3-propane tricarboxylates described in GB-A-1,387,447.

5

Polycarboxylate containing four carboxy groups include oxydisuccinates disclosed in GB-A-1,261,829, 1,1,2,2-ethane tetracarboxylates, 1,1,3,3-propane tetracarboxylates and 1,1,2,3-propane tetracarboxylates.

10 Polycarboxylates containing sulfo substituents include the sulfosuccinate derivatives disclosed in GB-A-1,398,421, GB-A-1,398,422 and US-A-3,936448, and the sulfonated pyrrolised citrates described in GB-A-1,439,000.

15 Alicyclic and heterocyclic polycarboxylates include cyclopentane-cis,cis,cis-tetracarboxylates, cyclopentadienide pentacarboxylates, 2,3,4,5,6-hexane - hexacarboxylates and carboxymethyl derivatives of polyhydric alcohols such as sorbitol, mannitol and
20 xylitol. Aromatic polycarboxylates include mellitic acid, pyromellitic acid and the phthalic acid derivatives disclosed in GB-A-1,425,343.

Of the above, the preferred polycarboxylates are
25 hydroxycarboxylates containing up to three carboxy groups per molecule, more particularly citrates.

More preferred are the polymer builders, i.e. polymeric polycarboxylic acid, which are homo-polymers,
30 copolymers and multiple polymers of acrylic, fluorinated acrylic, sulfonated styrene, maleic anhydride, metacrylic, iso-butylene, styrene and ester monomers. Examples of these polymers are Acusol supplied from Rohm & Haas, Syntran supplied from Interpolymer and Versa and

Alcosperse series supplied from Alco Chemical, a National Starch & Chemical Company.

Suitable builders are bicarbonates,
5 sesquicarbonates, borates, phosphates, phosphonates, and mixtures of any of thereof.

Water-soluble phosphonate and phosphate builders are useful for this invention. Examples of phosphate builders
10 are the alkali metal tripolyphosphates, sodium potassium and ammonium pyrophosphate, sodium and potassium and ammonium pyrophosphate, sodium and potassium orthophosphate sodium polymeta/phosphate in which the degree of polymerisation ranges from 6 to 21, and salts
15 of phytic acid.

Specific examples of water-soluble phosphate builders are the alkali metal tripolyphosphates, sodium potassium and ammonium pyrophosphate, sodium and
20 potassium and ammonium pyrophosphate, sodium and potassium orthophosphate, sodium polymeta/phosphate in which the degree of polymerization ranges from 6 to 21, and salts of phytic acid.

25 Examples of bicarbonate and carbonate builders are the alkaline earth and the alkali metal carbonates, including sodium carbonate and sesqui-carbonate and mixtures thereof. Other examples of carbonate type builders are the metal carboxy glycine and metal glycine
30 carbonate.

Surfactants

Examples of surfactants considered in this invention are either anionic, non-ionic or cationic. Preferred total levels of surfactant are from 0.1 to 70% w/w, ideally from 1 to 30% wt and preferably between 5 to 5 20%w/w.

Examples of non-ionic surfactant are described in the formula $RO(CH_2CH_2O)_nH$ wherein R is a mixture of linear, even carbon-number hydrocarbon chains ranging 10 from $C_{12}H_{25}$ to $C_{16}H_{33}$ and n represents the number of repeating units and is a number of from about 1 to about 12. Examples of other non-ionic surfactants include higher aliphatic primary alcohols containing about twelve to about 16 carbon atoms which are condensed with about 15 three to thirteen moles of ethylene oxide.

Other examples of non-ionic surfactants include primary alcohol ethoxylates (available under the Neodol tradename from Shell Co.), such as C_{11} alkanol condensed 20 with 9 moles of ethylene oxide (Neodol 1-9), C_{12-13} alkanol condensed with 6.5 moles ethylene oxide (Neodol 23-6.5), C_{12-13} alkanol with 9 moles of ethylene oxide (Neodol 23-9), C_{12-15} alkanol condensed with 7 or 3 moles ethylene oxide (Neodol 25-7 or Neodol 25-3), C_{14-15} 25 alkanol condensed with 13 moles ethylene oxide (Neodol 45-13), C_9-11 linear ethoxylated alcohol, averaging 2.5 moles of ethylene oxide per mole of alcohol (Neodol 91-2.5), and the like.

30 Other examples of non-ionic surfactants suitable for use in the present invention include ethylene oxide condensate products of secondary aliphatic alcohols containing 11 to 18 carbon atoms in a straight or branched chain configuration condensed with 5 to 30 moles

of ethylene oxide. Examples of commercially available non-ionic detergents of the foregoing type are C11-15 secondary alkanol condensed with either 9 moles of ethylene oxide (Tergitol 15-S-9) or 12 moles of ethylene oxide (Tergitol 15-S-12) marketed by Union Carbide, a subsidiary of Dow Chemical.

Octylphenoxy polyethoxyethanol type non-ionic surfactants, for example, Triton X-100, as well as amine oxides can also be used as a non-ionic surfactant in the present invention.

Other examples of linear primary alcohol ethoxylates are available under the Tomadol tradename such as, for example, Tomadol 1-7, a C11 linear primary alcohol ethoxylate with 7 moles EO; Tomadol 25-7, a C12-C15 linear primary alcohol ethoxylate with 7 moles EO; Tomadol 45-7, a C14-C15 linear primary alcohol ethoxylate with 7 moles EO; and Tomadol 91-6, a C9-C11 linear alcohol ethoxylate with 6 moles EO.

Amine oxides can also be used as the non-ionic surfactant of the present invention. Exemplary useful amine oxide compounds may be defined as one or more of the following of the four general classes:

(1) Alkyl di (lower alkyl) amine oxides in which the alkyl group has about 6-24, and preferably 8-18 carbon atoms, and can be straight or branched chain, saturated or unsaturated. The lower alkyl groups include between 1 and 7 carbon atoms, but preferably each include 1 - 3 carbon atoms. Examples include octyl dimethyl amine oxide, lauryl dimethyl amine oxide, myristyl dimethyl amine oxide, and those in which the alkyl group is a mixture of different amine oxides, such as dimethyl

cocoamine oxide, dimethyl (hydrogenated tallow) amine oxide, and myristyl/palmityl dimethyl amine oxide;

(2) Alkyl di (hydroxy lower alkyl) amine oxides in which the alkyl group has about 6-22, and preferably 8-18 carbon atoms, and can be straight or branched chain, saturated or unsaturated. Examples include bis-(2-hydroxyethyl) cocoamine oxide, bis(2-hydroxyethyl) tallowamine oxide; and bis-(2-hydroxyethyl) stearylamine oxide;

(3) Alkylamidopropyl di(lower alkyl) amine oxides in which the alkyl group has about 10-20, and preferably 12-16 carbon atoms, and can be straight or branched chain, saturated or unsaturated. Examples include cocoamidopropyl dimethyl amine oxide and tallowamidopropyl dimethyl amine oxide; and

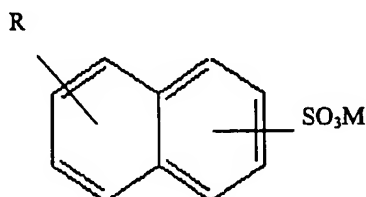
(4) Alkylmorpholine oxides in which the alkyl group has about 10-20, and preferably 12-16 carbon atoms, and can be straight or branched chain, saturated or unsaturated.

20

Useful anionic surfactant are frequently provided in a salt form, such as alkali metal salts, ammonium salts, amine salts, amino alcohol salts or magnesium salts. Contemplated as useful are one or more sulfate or sulfonate compounds including: alkyl sulfates, alkyl ether sulfates, alkylamidoether sulfates, alkyl benzene sulfates, alkyl benzene sulfonates, alkylaryl polyether sulfates, monoglyceride sulfates, alkylsulfonates, alkylamide sulfonates, alkylarylsulfonates, olefinsulfonates, paraffin sulfonates, alkyl sulfosuccinates, alkyl ether sulfosuccinates, alkylamide sulfosuccinates, alkyl sulfosuccinamate, alkyl sulfoacetates, alkyl carboxylates, alkyl phosphates, alkyl ether phosphates, acyl sarconsinates, acyl

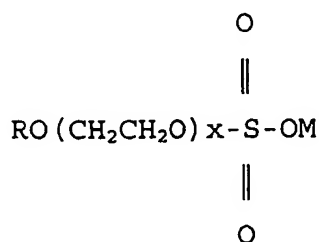
isethionates, and N-acyl taurates. Generally, the alkyl or acyl radical in these various compounds comprise a carbon chain containing 12 to 20 carbon atoms.

Other examples of anionic surfactants are also alkyl naphthalene sulfonate anionic surfactants of the formula:



wherein R is a straight chain or branched alkyl chain having from about 1 to about 25 carbon atoms, saturated or unsaturated, and the longest linear portion of the alkyl chain is 15 carbon atoms or less on the average, M is a cation which makes the compound water soluble especially an alkali metal such as sodium or magnesium, ammonium or substituted ammonium cation.

Other examples are alkyl sarcosinate, sulfosuccinate and alkyl sulfate anionic surfactants of the formula

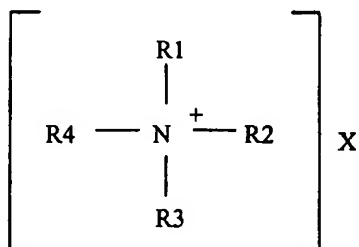


wherein R is a straight chain or branched alkyl chain having from about 8 to about 18 carbon atoms, saturated or unsaturated, and the longest linear portion of the alkyl chain is 15 carbon atoms or less on the average, M is a cation which makes the compound water soluble

especially an alkali metal such as sodium or magnesium, ammonium or substituted ammonium cation, and x is from 0 to about 4. Most preferred are the non-ethoxylated C12-15 primary and secondary alkyl sulfates, especially sodium lauryl sulfate.

Most desirably, the anionic surfactant according to constituent is selected to be of a type that dries to a friable powder. This facilitates their removal from carpets and carpet fibres, such as by brushing or vacuuming.

The cationic surfactants of the invention are quaternary ammonium salts which may be characterised by the general structural formula:



wherein R1, R2, R3 and R4 are independently selected from alkyl, aryl or alkylaryl substituent of from 1 to 26 carbon atoms, and the entire cation portion of the molecule has a molecular weight of at least 165. The alkyl substituents may be long-chain alkyl, long-chain alkoxyaryl, long-chain alkylaryl, halogen-substituted long-chain alkylaryl, long-chain alkylphenoxyalkyl and arylalkyl. The remaining substituents on the nitrogen atoms other than the above mentioned alkyl substituents are hydrocarbons usually containing no more than 12 carbon atoms. The substituents R1, R2, R3 and R4 may be straight-chained or may be branched, but are preferably

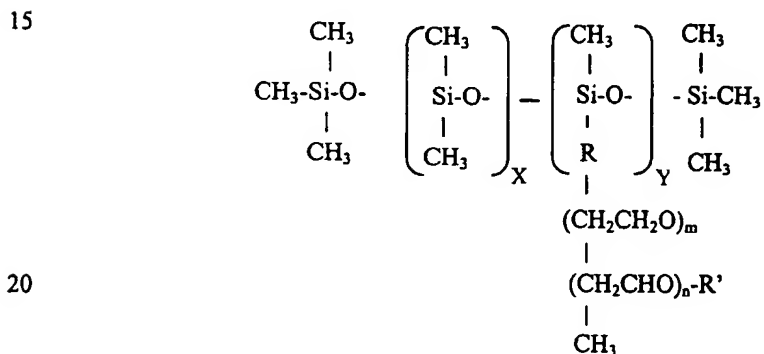
straight-chained, and may include one or more amide, ether or ester linkages.

The counterion X- are selected from halogens anions, saccharinate, alkyl and alkyl benzene sulfate, sulfonate
5 and fatty acid.

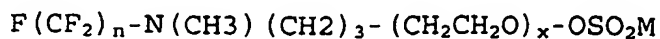
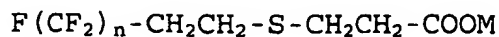
Super wetting agents

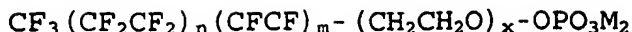
The super wetting agents of this invention are
10 present at levels of from 0.1 to 10% w/w, ideally 0.5 to 5% w/w, and are selected from silicone glycol copolymers and fluorsurfactants.

The silicone glycol copolymers are described by the following formula:



Where X, Y, m and n are whole number ranging from 0 to 25. X is preferably between 0 to 10 and Y, m and n
25 between 0 to 5. R and R' are straight chain or branched alkyl chain having from about 1 to 25 carbon atoms, saturated or unsaturated, and the longest linear portion of the alkyl chain is on average 15 carbon atoms or less. The fluorinated surfactant is described in the following
30 formulae:





Wherein n, m and x are integers having a value from 0 to 15; preferred values are between 1 and 12. M is a cation
5 which is capable of making the compound water-soluble, especially an alkali metal such as sodium or magnesium or an ammonium or substituted ammonium cation.

The super wetting agents described are able to lower
10 the surface tension in water at values below 25 mN/m at a concentration less than 0.1% w/v.

Antifoaming

15 Antifoaming agents are an important addition to carpet cleaning compositions of this invention, they are used at a level between 0.01 and 5%w/w. A very high foam level may not allow the carpet cleaning machine to function properly. Antifoaming agents are also considered
20 important components of this invention. Examples are polydimethylsiloxanes, preferably in combination with hydrophobic silica.

Solvents:

25

Organic solvents should be water-miscible or water emulsionable. The organic solvent is found at levels of 0.01 to 60% w/v, more preferably between 0.1 to 30% w/w. The organic solvent constituent of the inventive
30 compositions include one or more alcohols, glycols, acetates, ether acetates, glycol ethers and hydrocarbons. Exemplary alcohols useful in the compositions of the invention include C2-C8 primary and secondary alcohols which may be straight chained or branched. Exemplary

alcohols include pentanol and hexanol. Exemplary glycol ethers include those glycol ethers having the general structure Ra-O-Rb-OH, wherein Ra is an alkoxy of 1 to 20 carbon atoms, or aryloxy of at least 6 carbon atoms, and
5 Rb is an ether condensate of propylene glycol and/or ethylene glycol having from 1 to 10 glycol monomer units. Preferred are glycol ethers having 1 to 5 glycol monomer units.

10 By way of further non-limiting example specific organic constituents include propylene glycol methyl ether, dipropylene glycol methyl ether, tripropylene glycol methyl ether, propylene glycol n-propyl ether, ethylene glycol n-butyl ether, diethylene glycol n-butyl
15 ether, diethylene glycol methyl ether, propylene glycol, ethylene glycol, isopropanol, ethanol, methanol, diethylene glycol monoethyl ether acetate and particularly useful is , propylene glycol phenyl ether, ethylene glycol hexyl ether, diethylene glycol hexyl
20 ether. Examples of hydrocarbons solvents are linear and branched, saturated and unsaturated carbon chain with a number of carbon atoms from C4-C40, preferably from C6-C22.

25 Process manufacturing:

In one embodiment, a compartment of water-soluble polymer is formed by moulding of a water-soluble polymer, especially one produced by injection moulding or blow
30 moulding, such as described in WO 0136290. The compartment may have a wall thickness of, for example, greater than 100 µm, for example greater than 150 µm or

greater than 200 μm , 300 μm , 500 μm , 750 μm or 1mm.
Preferably, the wall thickness is from 200 to 400 μm .

In an alternative embodiment the compartment may,
5 for example, be formed of a film of the water-soluble
polymer. The film may be a single film, or a laminated
film as disclosed in GB-A-2,244,258. While a single film
may have pinholes, the two or more layers in a laminate
are unlikely to have pinholes that coincide.

10

The film may be produced by any process, for example
by extrusion and blowing or by casting. The film may be
unoriented, monoaxially oriented or biaxially oriented.
If the layers in the film are oriented, they usually have
15 the same orientation, although their planes of
orientation may be different if desired.

The layers in a laminate may be the same or
different. Thus they may each comprise the same polymer
20 or a different polymer.

Examples of water-soluble polymers which may be used
in a single layer film or in one or more layers of a
laminate or which may be used for injection moulding or
25 blow moulding are poly(vinyl alcohol) (PVOH), cellulose
derivatives such as hydroxypropyl methyl cellulose (HPMC)
and gelatin. An example of a preferred PVOH is
ethoxylated PVOH. The PVOH may be partially or fully
alcoholised or hydrolysed. For example it may be from 40
30 to 100%, preferably from 70 to 92%, more preferably about
88% or about 92%, alcoholised or hydrolysed. The degree
of hydrolysis is known to influence the temperature at
which the PVOH starts to dissolve in water. 88%

hydrolysis corresponds to a film soluble in cold (ie room temperature) water, whereas 92% hydrolysis corresponds to a film soluble in warm water.

5 The thickness of the film used to produce the container, is preferably 40 to 300 μm , more preferably 80 to 200 μm , especially 100 to 160 μm , more especially 100 to 150 μm and most especially 120 to 150 μm .

10 In one embodiment, the film is vacuum formed or thermoformed into a compartment for the first composition, such as described in WO 0216207 For example, in a thermoforming process the film may be drawn down or blown down into a mould. Thus, for example, the film is
15 heated to the thermoforming temperature using a thermoforming heater plate assembly, and then drawn down under vacuum or blown down under pressure into the mould. Plug-assisted thermoforming and pre-stretching the film, for example by blowing the film away from the mould
20 before thermoforming, may, if desired, be used. One skilled in the art can choose an appropriate temperature, pressure or vacuum and dwell time to achieve an appropriate shape. The amount of vacuum or pressure and the thermoforming temperature used depend on the
25 thickness and porosity of the film and on the polymer or mixture of polymers being used. Thermoforming of PVOH films is known and described in, for example, WO 00/55045 and WO 01/85898.

30 A suitable forming temperature for PVOH or ethoxylated PVOH is, for example, from 90 to 130°C, especially 90 to 120°C. A suitable forming pressure is, for example, 69 to 138kPa (10 to 20 p.s.i.), especially

83 to 117 kPa (12 to 17 p.s.i.). A suitable forming vacuum is 0 to 4 kPa (0 to 40 mbar), especially 0 to 2 kPa (0 to 20 mbar). A suitable dwell time is, for example, 0.4 to 2.5 seconds, especially 2 to 2.5 seconds.

5

While desirably conditions chosen within the above ranges, it is possible to use one or more of these parameters outside the above ranges, although it may be necessary to compensate by changing the values of the other two parameters.

Once formed, the compartment may be filled with the liquid carpet cleaning composition. The compartment may be completely filled or only partially filled. The liquid composition may be thickened or gelled, if desired. More preferably, the composition is a transparent liquid, especially, a coloured, transparent liquid. The liquid composition may be non-aqueous or aqueous, although preferably comprising less than 5% free water as defined in WO 02/16222. The composition may have more than one phase. For example it may comprise an aqueous composition and a liquid composition that is immiscible with the aqueous composition. It may also comprise a liquid composition and a separate solid composition, for example in the form of a ball, pill or speckles.

The compartment is sealed with a sealing member and may be sealed together by any suitable means, for example by means of an adhesive or by heat sealing. Mechanical means is particularly appropriate if both have been prepared by injection moulding. Other methods of sealing include infra-red, radio frequency, ultrasonic, laser, solvent, vibration and spin welding. An adhesive such as an aqueous solution of PVOH may also be used. The seal

desirably is water-soluble if the containers are water-soluble.

If heat sealing is used, a suitable sealing temperature is, for example, 120 to 195°C, for example 140 to 150°C. A suitable sealing pressure is, for example, from 250 to 600 kPa. Examples of sealing pressures are 276 to 552 kPa (40 to 80 p.s.i.), especially 345 to 483 kPa (50 to 70 p.s.i.) or 400 to 800 kPa (4 to 8 bar), especially 500 to 700 kPa (5 to 7 bar) depending on the heat sealing machine used. Suitable sealing dwell times are 0.4 to 2.5 seconds.

One sealing member is made also from a water-soluble polymer, although not necessarily of the same water-soluble polymer as the compartment. The sealing member may be a film or a moulded piece.

One skilled in the art can use an appropriate temperature, pressure and dwell time to achieve a seal of the desired integrity. While desirably conditions are chosen within the above ranges, it is possible to use one or more of these parameters outside the above ranges, although it would might be necessary to compensate by changing the values of the other two parameters.

The containers may themselves be packaged in outer containers if desired, for example non-water soluble containers which are removed before the water-soluble containers are used.

The containers produced by the process of the present invention, may have a maximum dimension of 5 cm, excluding any flanges. For example, a container may have

desirably is water-soluble if the containers are water-soluble.

If heat sealing is used, a suitable sealing
5 temperature is, for example, 120 to 195°C, for example
140 to 150°C. A suitable sealing pressure is, for
example, from 250 to 600 kPa. Examples of sealing
pressures are 276 to 552 kPa (40 to 80 p.s.i.),
especially 345 to 483 kPa (50 to 70 p.s.i.) or 400 to 800
10 kPa (4 to 8 bar), especially 500 to 700 kPa (5 to 7 bar)
depending on the heat sealing machine used. Suitable
sealing dwell times are 0.4 to 2.5 seconds.

One sealing member is made also from a water-soluble
15 polymer, although not necessarily of the same water-
soluble polymer as the compartment. The sealing member
may be a film or a moulded piece.

One skilled in the art can use an appropriate
20 temperature, pressure and dwell time to achieve a seal of
the desired integrity. While desirably conditions are
chosen within the above ranges, it is possible to use one
or more of these parameters outside the above ranges,
although it would might be necessary to compensate by
25 changing the values of the other two parameters.

The containers may themselves be packaged in outer
containers if desired, for example non-water soluble
containers which are removed before the water-soluble
30 containers are used.

The containers produced by the process of the
present invention, may have a maximum dimension of 5 cm,
excluding any flanges. For example, a container may have

a length of 1 to 5 cm, especially 3.5 to 4.5 cm, a width of 1.5 to 3.5 cm, especially 2 to 3 cm, and a height of 1 to 2 cm, especially 1.25 to 1.75 cm.

5 SURFACE TENSION MEASUREMENTS:

The surface tension has been measured with a tensiometer, by the ring method. A platinum du Nuoy ring is immersed in 12 French degree water solution maintained at 20°C, containing a dissolved capsule of the invention. The ring is taken out slowly from the liquid. When the ring is near the air/liquid interface, it is balanced by a tensiometer to measure the force for the ring extraction. The maximum force versus area gained before breaking the liquid film formed is the surface tension of the liquid formula.

The lower the surface tension expressed in mN/m, the better the formula performance in terms of wettability on carpet surface.

20 A surface tension target value for the composition is below 28 mN/m.

Evaluation Test

The compositions have been evaluated in terms of storage stability, dissolution time in warm water (T=40°C), remaining residue after dissolution and soil removal performance versus Resolve™ for steam machine taken as a reference of the machine carpet cleaner products.

30

STORAGE STABILITY TEST:

Two containers for each condition were stored at 5°C, 25°C and 50°C in climatic cells. The change in the

appearance of the container and the weight changes are monitored after 2 weeks, 1 month, 3 months and 6.

A recording data table with the corresponding storage stability rating is reported below:

5

Storage rating	Liquid appearance	Film appearance	Weight loss (g)
0	No separation	No difference from reference	No weight loss
1	No separation	Slightly humid and flexible	Less than 1% weight loss
2	Separation	Slightly humid and flexible	Less than 1% weight loss
3	No separation	Slightly humid and flexible	Weight loss between 1-5%
4	Separation	Slightly humid and flexible	Weight loss between 1-5%
5	No separation	Humid and flexible	More than 5% weight loss
6	Separation	Humid and flexible	More than 5% weight loss
7	No separation	Very humid and flexible	More than 5% weight loss
8	Separation	Very humid and flexible	More than 5% weight loss
9	-	The film is broken -	-

The higher the rating number then the worse is the stability of the composition.

10 DISSOLUTION AND RESIDUE EVALUATION TEST:

One container of 8 ml of composition is dissolved in a beaker containing $\frac{1}{2}$ gallon of warm water ($T=40^{\circ}\text{C}$) with stirring and without stirring. The dissolution time as well as the remaining film residue quantity are recorded.

5

Dissolution rating	Dissolution time	Film residue percentage
0	More than 30 minutes	100% wt
1	Between 10 - 30 minutes	Between 20% wt
2	More than 10 minutes	More than 10% wt
3	Between 5 and 10 minutes	More than 10% wt
4	Between 5 and 10 minutes	Between 5 and 10 % wt
5	Between 5 and 10 minutes	Between 5 and 10 % wt
6	Less than 5 minutes	Between 5 and 10 % wt
7	Less than 5 minutes	Less than 5 % wt

The higher the rating number then the better is the dissolution behaviour of the container.

10 SOIL REMOVAL TEST:

This method has been designed for the evaluation of soil removal performance of extraction cleaner formulations.

15

The soil has the following composition:

	- Soil components:	% by weight
	- Peat Moss	47.7
	- Cement	21.4
5	- Kaolin clay	8.0
	- Silica	8.0
	- Red Iron oxide	1.3
	- Charcoal	12.6
	- Mineral oil	1.0

10

A nylon carpet is used for the test.

The carpet is soiled with 5 grams of standard soil. The soil is applied 1 gram once by strainer. The soiled
15 carpet is then put with 4 kg of steel beads in a jar mill and stirred for 30 minutes at 56 rpm.

The containers are dissolved in warm tap water (T=40°C). The Resolve for steam machine is diluted
20 according to its labelling instructions.

Carpet is cleaned with appropriate machine/product using 4 wet strokes (dispensing solution) and 2 dry strokes (vacuuming up solution). Carpet swatches are
25 placed in a dark room temperature chamber (25°C/50%RH) for 24 hours while they dry.

The cleaning performance is evaluated by measuring the carpet with a portable spectrophotometer before
30 soiling, after soiling and after the cleaning process. The result is reported as soil removal percentage versus current Resolve steam machine liquid taking 100 as a reference.

EXAMPLES:

The liquids are typically prepared by mixing all the components together in a suitable container to form a concentrate. The liquid concentrates are then filled in PVOH film pockets and heat sealed by using a Magic Vac Elite machine. The heat sealing process is carried out at 150°C and 1 bar pressure. PVOH films used in the present invention are L712D obtained from Aquafilm ltd, UK) having a thickness of 120µm and M8630 obtained from Chris Craft having a thickness of 76µm

Examples of compositions forming a part of the present invention are set forth below in Table 1 and 2; with various components identified in Table 3.

Components	<u>Table 1</u>				
	Ex 1	Ex 2	Ex 3	Ex 4	Ex 5
	%	%	%	%	%
EDTA	71.98	71.98			71.63
Trilon B			37.41		
Trilon BS acid			3.54		
Citric acid	5.88	5.88		0.63	5.63
Petro 11 powder	14.70	14.70	5.67	33.13	15.00
Nonionic			9.36	3.13	
Syntran 1580			7.33		
Syntran 4015				62.50	
Dowanol PnP			34.60		
Propylene glycol	5.94	7.44			
PEG 400					6.25
Fragrance	1.50		1.85	0.63	1.50
Antifoam			0.23		
Water added*	0.00	0.00	0.00	0.00	0.00

Water content	47.04	47.04	27.33	60.31	46.80
---------------	-------	-------	-------	-------	-------

	<u>Table 2</u>				
Components	Ex 6	Ex 7	Ex 8	Ex 9	Ex 10
	%	%	%	%	%
Baypure CX100/34	37.50		50.00	63.75	62.62
Baypure DS100/40	8.22		4.38	7.88	7.66
Trilon B	3.75	32.44			
Trilon BS acid		3.07			
Citric acid			3.75	5.63	5.55
Petro 11 powder	15.62	4.92	18.75	15.00	14.14
Genapol 26-L-60	2.06	8.13			
Syntran 1580		6.50			
Propylene glycol	31.25	13.15	21.63	6.25	6.13
M-Pyrol		30.00			
Fragrance	1.60	1.60	1.50	1.50	1.43
Antifoam		0.20			
Aerosil 200					2.46
Water added*	0.00	0.00	0.00	0.00	0.00
Water content	31.34	23.93	35.63	46.80	47.38

*Some water is contained in some of the raw materials, such as CX 100/34, DS100/40, Syntran 1580, SE21 and

5 Trilon B.

	<u>Table 3</u>
Component	Description of component
Baypure CX100/34	Iminosuccinic sodium salt from Bayer
Baypure DS100/40	Polyaspartic acid sodium salt from
Petro 11 powder	Alkyl naphthalene sulfonate from Witco
M-Pyrol	1-methyl 2-pyrrolidinone from ISP
Aerosil 200 powd.	Amorphous silica from Degussa
Syntran 1580	Acrylic copolymer from Interpolymer
Syntran 4015	Acrylic copolymer from Interpolymer

Dowanol PnF	1-Propoxy-2-propanol from Dow chem
PEG 400	Polyethylene glycol from Basf

EXAMPLE RESULTS:

The products were tested in terms of storage stability,
5 dissolution time and soil removal performance..

Results for storage stability test:

8 ml gel caps have been used for storage stability.

10

Product	Stability test rating											
	2 weeks			1 month			3 months			6 months		
	5°C	25° C	50° C	5°C	25° C	50° C	5°C	25° C	50° C	5°C	25° C	50° C
Ex 1	1	1	1	1	1	3	1	3	3	2	5	5
Ex 2	0	1	1	0	1	3	1	5	5	1	5	9
Ex 3	3	9	9	-	-	-	-	-	-	-	-	-
Ex 4	8	9	9	-	-	-	-	-	-	-	-	-
Ex 5	2	5	5	2	9	9	-	-	-	-	-	-
Ex 6	5	9	9	-	-	-	-	-	-	-	-	-
Ex 7	9	9	9	-	-	-	-	-	-	-	-	-
Ex 8	1	1	3	1	3	3	1	5	5	1	8	8
Ex 9	1	1	1	1	1	1	1	3	3	1	3	3
Ex 9	1	9	9	-	-	-	-	-	-	-	-	-
Ex 10	1	1	1	1	1	2	1	1	2	1	1	2

Results for dissolution test:

Product	Dissolution test	
	with stirring	without stirring

Ex 9	6	3	5	2
Ex 10	6	-	6	-

Results for soil removal test:

The data are compared with the Resolve for steam machine
5 liquid formulation taken as a reference of 100 of soil
removal. The Resolve for steam machine product has been
diluted 4 oz/gall according to its labelling instruction.

Product	Soil removal test	
	1 gel cap/gall	2 gel caps/gall
Ex 1	89	-
Ex 2	96	-
Ex 8	61	-
Ex 9	145	120
Ex 10	135	-

CLAIMS

1. A method of cleaning a soiled carpet the method comprising adding to the reservoir of a carpet cleaning machine a water soluble product comprising a liquid carpet cleaning composition encased in a water soluble polymer, adding water to the reservoir of the carpet cleaning machine prior to or after adding the water soluble product, and operating the carpet cleaning machine on the soiled carpet.
2. A method as claimed in claim 1 wherein the water soluble product is added to the reservoir of the carpet cleaning machine prior to adding the water.
3. A method as claimed in claim 2 or claim 3 wherein the liquid carpet cleaning composition contains up to 50%w/w of free water.
4. A method as claimed in claim 3 wherein the composition additionally contains at least one builder.
5. A method as claimed in any claim from 1 to 4 wherein the carpet cleaning composition comprises at least one surfactant and one super wetting agent and wherein the combined effect of the surfactant and the super wetting agent in the composition is capable of reducing the surface tension of water below 28 mN/m when 10g of the carpet cleaning composition is dissolved in 4 litres of water.
6. A water-soluble carpet cleaning products comprising a liquid carpet cleaning composition encased in a

water-soluble polymer the liquid carpet cleaning composition having at least one surfactant and optionally one super wetting agent wherein the combined surface tension effect of the surfactant and the super wetting agent in the composition is capable of reducing the surface tension of water below 28 mN/m when 10g of the liquid carpet cleaning composition is dissolved in 4 litres of water

10 7. A water-soluble carpet cleaning product as claimed in claim 6, wherein carpets cleaning liquid composition additionally comprises an antifoaming agent, a solvent, a thickener, a dye and a fragrance.

15

8. A water-soluble carpet cleaning product as claimed in any claim from 6 to 8 wherein the water-soluble polymer is poly(vinyl alcohol).

INTERNATIONAL SEARCH REPORT

International Application No.

PCT/GM/04329

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C11D11/00 C11D17/04 C11D1/00 C11D3/37 A47L11/34

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C11D A47L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 01 83667 A (PROCTER & GAMBLE) 8 November 2001 (2001-11-08) claims examples	6-8
A	US 5 728 669 A (TYERECH MICHAEL RICHARD) 17 March 1998 (1998-03-17) claims examples column 8, line 11 - line 26 column 9, line 16 - line 55	1-8
A	WO 98 47358 A (BLACK ROBERT H) 29 October 1998 (1998-10-29) claims page 3, line 18 -page 4, line 5 page 5, line 18 -page 6, line 2	1-8

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- * & * document member of the same patent family

Date of the actual completion of the international search

2 February 2004

Date of mailing of the international search report

11/02/2004

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Neys, P

INTERNATIONAL SEARCH REPORT

ion on patent family members

Internatic

plication No

PCT/G 3/04329

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
WO 0183667	A	08-11-2001	GB 2361688 A	31-10-2001
			AU 4356201 A	12-11-2001
			AU 4735901 A	12-11-2001
			BR 0110271 A	18-02-2003
			BR 0110322 A	07-01-2003
			CA 2404681 A1	08-11-2001
			CA 2404689 A1	08-11-2001
			CN 1426458 T	25-06-2003
			CN 1426459 T	25-06-2003
			EP 1283862 A1	19-02-2003
			EP 1276846 A2	22-01-2003
			JP 2003531951 T	28-10-2003
			JP 2003531953 T	28-10-2003
			WO 0183667 A1	08-11-2001
			WO 0183658 A2	08-11-2001
			US 2003087783 A1	08-05-2003
			US 2003092590 A1	15-05-2003

US 5728669	A	17-03-1998	GB 2321251 A	22-07-1998
			AU 737491 B2	23-08-2001
			AU 4826897 A	07-08-1998
			BR 9714204 A	28-03-2000
			DE 69724831 D1	16-10-2003
			EP 0971998 A1	19-01-2000
			WO 9831777 A1	23-07-1998
			ZA 9800202 A	13-07-1998

WO 9847358	A	29-10-1998	AU 7254798 A	13-11-1998
			EP 0977481 A1	09-02-2000
			JP 2001522359 T	13-11-2001
			US 6239166 B1	29-05-2001
			WO 9847358 A1	29-10-1998
